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EXPERIMENTAL STUDY OF THE COATING FOUND ON SCUFFED CAST-IRON  
PISTON RINGS DURING OPERATION IN NITRIDED-STEEL CYLINDERS

By J. Howard Kittel and Walter A. Vierthaler

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ADVANCE CONFIDENTIAL REPORT

EXPERIMENTAL STUDY OF THE COATING FOUND ON SCUFFED CAST-IRON  
PISTON RINGS DURING OPERATION IN NITRIDED-STEEL CYLINDERS

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SUMMARY

Cast-iron piston rings that were run in nitrided-steel cylinder barrels under scuffing conditions showed a coating material on the scuffed areas. Only a few of the 14 etching reagents that were tried attacked the coating chemically, indicating good corrosion resistance. The Vickers hardness number of the coating material was found to be 640. The coating formed to a maximum thickness of about 0.0001 inch on the running surface of the piston rings. Evidence was found that steadite in the cast iron is associated with the formation of the coating. The coating also appears to contain voids or minute flakes of graphite. On the basis of results obtained from the use of Stead's reagent, it was found that the surfaces of cast-iron piston rings that have been run under normal operating conditions show more steadite than new rings and that scuffed rings show more steadite than either new or normally operated unscuffed rings.

INTRODUCTION

Cast-iron piston rings show differences in surface structure before and after running-in: run-in cast-iron rings may have a hard surface and often possess a glazed appearance; the surface of as-machined rings is no harder than the basis metal and usually possesses a dull matte appearance. The run-in surface is of great importance because of the ability of run-in rings to carry large sliding loads with little or no damage to the sliding surfaces. The run-in surface is sometimes called the Beilby layer because of the theoretical similarity between the run-in surface and that produced by Sir George Beilby in polishing several materials, notably calcite (reference 1). Polish layers in general have never been seen in cross section with the optical microscope because of their extreme thinness, which is estimated to be from 30 to 600 Å (references 2 and 3). Electron-diffraction methods, however, yield information enabling the presence of polish layers to be detected.

Run-in layers, which are distinct from polish layers because they are produced under actual or simulated operating conditions of the sliding members, have been examined, both in the United States and abroad, on several materials such as cast iron, stainless steel, and spring steel (reference 4). These layers are ordinarily of sufficient thickness to be visible under the optical microscope.

Studies of coatings on surfaces of sliding metals have been published by J. T. Burwell, Jr. and J. Wulff (reference 5), C. S. Lees (reference 6), and W. Cochrane (reference 7).

The fact that run-in cast-iron rings are capable of operating satisfactorily under adverse conditions of sliding indicates that the run-in properties of the surface are produced by a local overloading sufficient to alter the surface structure in spots but not so widespread as to overload large parts of the surface and thus produce failure. An investigation was conducted at the NACA Cleveland laboratory to determine whether cast-iron rings that had been run under conditions so severe as to produce scuffing might furnish an indication of the structure of the run-in layer on cast iron by showing an altered surface stratum so thick as to be microscopically visible. The coating layer herein described was observed by microscopically examining cast-iron piston rings that were run in nitrided-steel cylinder barrels. It is not claimed that the coating found on scuffed cast-iron rings is also found on unscuffed, well-run cast-iron rings.

Photomicrographs of piston-ring sections are presented to illustrate the physical structure of the coating material, to show the effects of various etching reagents on the material, and to indicate the probable origin and process of formation of the material.

#### APPARATUS

An inverted metallurgical microscope and camera were used for all photomicrographs at magnifications of 35 and 1500 diameters. Vertical bright-field illumination was used except where otherwise noted.

Two types of metallurgical section were made of the ring specimens: the first type was a cross section through the running face of the specimen; the second type of section was made by a taper-sectioning method mentioned in reference 8. The effective vertical magnification of taper sections was 25 times the actual horizontal magnification. All specimens were cleaned in organic solvents, plated with nickel in order to prevent the surface profiles of the specimens

from being rounded out of the plane of the section during polishing, and metallurgically polished. The etching reagents used are noted in each figure and are listed in table 1.

An indentation-type microhardness tester with a Vickers-type diamond indenter was employed to obtain the microhardness of the coating material. The indenter was loaded by a 25-gram spring force.

### TESTS AND RESULTS

The cast-iron piston rings that were studied in this investigation had been operated in nitrided-steel cylinder barrels under various conditions of service in single-cylinder engine tests conducted at Langley Field, Va. The tests were run at an engine speed of 2500 rpm and brake mean effective pressures of 210 to 250 pounds per square inch. The running time varied from  $3/4$  to  $9\frac{1}{2}$  hours.

A photomicrograph of a transverse section of a new cast-iron piston ring is presented, for convenience of reference, in figure 1. The structure consists of a number of constituents randomly distributed over the cross section. The cross section shows the presence of graphite, pearlite, and steadite.

### Occurrence of Coating

The coating present on scuffed cast-iron rings was not seen anywhere on the surface except at the scuffed regions. The entire scuffed area did not, however, show the presence of the coating, possible because of the extreme thinness of the layer at some points. The scuffed portion of the face of a cast-iron piston ring is ridged, as is shown in figure 2. Photomicrographs of high magnification, such as the one shown in figure 3, indicate that the coating, as a rule, tended to fill hollows in the surface profile of the basis metal.

Because the boundary between the coating and the basis metal in many places is not clearly definable, and because the basis structure appears to protrude into the coating material itself, it seems probable that the coating originates from the piston-ring constituents rather than from the cylinder barrel.

Figure 4 is a photomicrograph of the same area as shown in figure 3 but the etching reagent employed in this case was potassium hydroxide, which chiefly attacked the coating material and the steadite. The uppermost surface of the coating, despite its appearance in figure 2, was fairly smooth. The surface finish of the

scuffed areas ranged from 20 to 30 microinches, rms as compared with a surface finish of from 6 to 10 microinches, rms on a normally operated unscuffed ring. The maximum thickness of coating observed to date in transverse sections is about 0.0001 inch.

### Metallurgical Characteristics

The coating material appears to have been formed in part from steadite that had been exposed at the surface and had flowed over the running face. The coating material and steadite exhibit about the same response to metallographic etching reagents. It is entirely possible, however, that metallurgical and chemical differences exist between the coating material and steadite as usually encountered in the ring. Although the microstructure typical of the steadite can be seen below the surface, the coating material itself and the steadite for some small distance below the surface do not manifest an optically resolvable structure. Voids or inclusions of graphite may be seen embedded at random in the coating. (See fig. 5.) The taper section shown in figure 6 indicates that the coating is far from uniformly distributed over the ring face.

### Chemical Characteristics

The coating material was darkened by a hot concentrated solution of potassium hydroxide applied to the cross section of the scuffed specimens, but nital (nitric acid plus alcohol) does not appear to have attacked it. Reagents that attack steadite attacked the coating. Table 1 lists the 14 etching reagents employed in attempts to determine the corrosion resistance of the coating material. The high resistance to corrosion by acids indicates that the coating should resist corrosion by acid products formed in engines. No spectroscopic analysis was attempted because past experience has indicated that thin coatings permit the spectroscopic spark to penetrate to the basis metal and obscure the effect of the coating.

Microhardness tests of the coating gave an average value of 640 Vickers hardness number, which is equivalent to Rockwell C-55. It is emphasized that this hardness value is only approximate because, in most areas, the coating was of sufficient thinness to allow the basis metal to influence the hardness reading.

## DISCUSSION OF RESULTS

The thin coatings observed on portions of scuffed cast-iron piston rings were not easily attacked by dilute nitric acid but were visible attacked by hot concentrated potassium hydroxide. Steadite showed approximately the same chemical behavior. A transverse section of a coated cast-iron piston ring etched with nital (fig. 7) shows steadite apparently merging into the coating. Figure 8 shows the same area as figure 7, but in this case the section has been etched with hot concentrated potassium hydroxide. The apparent merging of the steadite into the coating is shown here even more clearly; it appears possible, therefore, that steadite is associated with the formation of the coating on scuffed cast iron.

The exact process that would permit steadite to form the coating may be envisioned when it is remembered that the iron-phosphorus eutectic is the lowest melting constituent of cast iron. Local frictional heating may cause the steadite to melt or at least become so plastic that it is easily flowed over the surface. This process is all the more probable because scuffing is held to be associated with high surface temperatures (reference 9) and the coating is visible on scuffed sections. Materials made up of very small crystals give rise to diffraction patterns that are diffuse (reference 10). If the coating consisted of very small crystals surrounding occasional flakes of graphite, the possible presence of flakes of graphite in the coating (see fig. 5) would account for the predominance of the graphite diffraction pattern found by others on run-in cast iron.

Figure 9 presents a plan view of a new cast-iron ring face that has been etched with Stead's reagent. On the basis of results obtained from the use of Stead's reagent, it appears that a large part of the face area is composed of steadite (which is the reflective or light portion of the surface). It seems likely then, that the machining of the piston ring may cause some steadite to flow over the surface. Figure 10 shows a piston ring that has been run but has not scuffed. There is more steadite in evidence on this ring than on the new ring in figure 9. The scuffed section of ring shown in figure 11 shows more steadite than either figures 9 or 10. This evidence, also, indicates that the coating is at least partly steadite.

## SUMMARY OF RESULTS

On the basis of evidence obtained from the use of metallographic etching reagents on cast-iron piston rings operated under scuffing conditions in single-cylinder engine tests, the following results were noted:

1. A coating was formed on much of the scuffed surface of cast-iron piston rings.

2. The coating on the particular rings investigated was not more than 0.0001 inch in thickness.

3. The coating contains voids or inclusions of graphite.

4. The coating is corrosion-resistant.

5. Steadite in the cast iron appears to be associated with the formation of the coating.

6. The use of Stead's reagent indicates that the faces of unscuffed cast-iron piston rings that have been run in show more steadite than new rings. Scuffed rings show more steadite than either new rings or unscuffed run-in rings.

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TABLE 1. - ETCHING REAGENTS USED AND THEIR  
EFFECT ON THE COATING MATERIAL

Reagent	Purpose	Effect
Nitric acid, hot and cold, dilute and concentrated	General corrosion	None
Sulfuric acid, hot and cold, dilute and concentrated	General corrosion	None
Hydrochloric acid, hot and cold, dilute and concentrated	General corrosion	None
Aqua regia	General corrosion	None
Oxidized nitric acid	General corrosion	None
Potassium hydroxide, hot concentrated	General corrosion	Darkened (steadite also darkened)
Sodium hydroxide, hot concentrated	General corrosion	Slightly darkened (steadite also slightly darkened)
Ammonium hydroxide	General corrosion	None
Stead's reagent	Deposits copper on areas low in phos- phorus	
Chromic acid and heat tinting	Darkens steadite	Darkened
Ammonium persulfate	Darkens ferrite	None
Sodium picrate, 25 percent	Darkens cementite	None
m-Nitrobenzene sulfonic acid	Darkens martensite	None
Stannous chloride	Chemical reduction	None

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*Nickel plate*

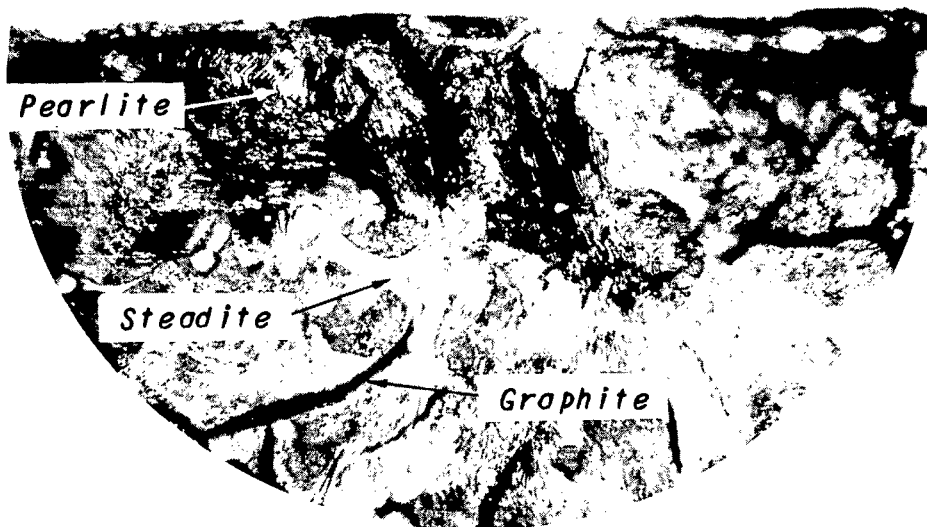
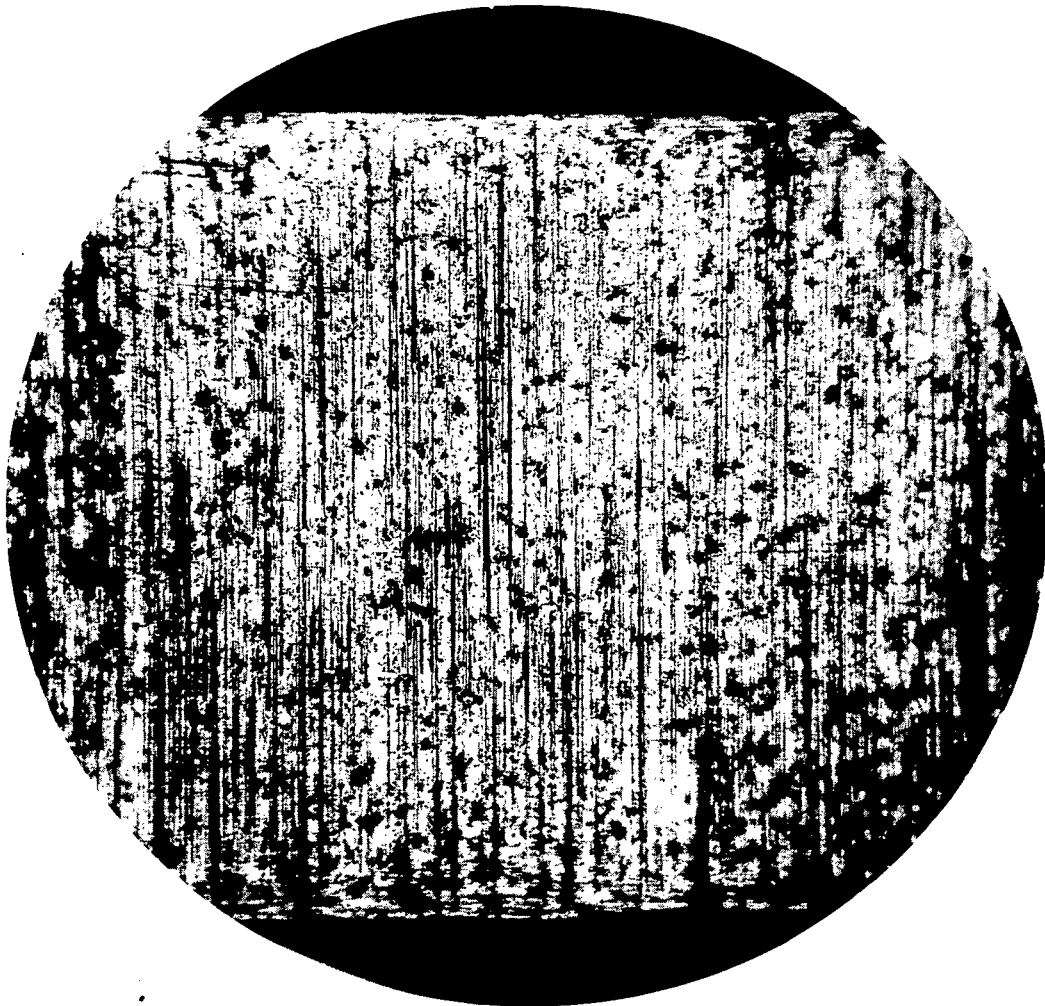
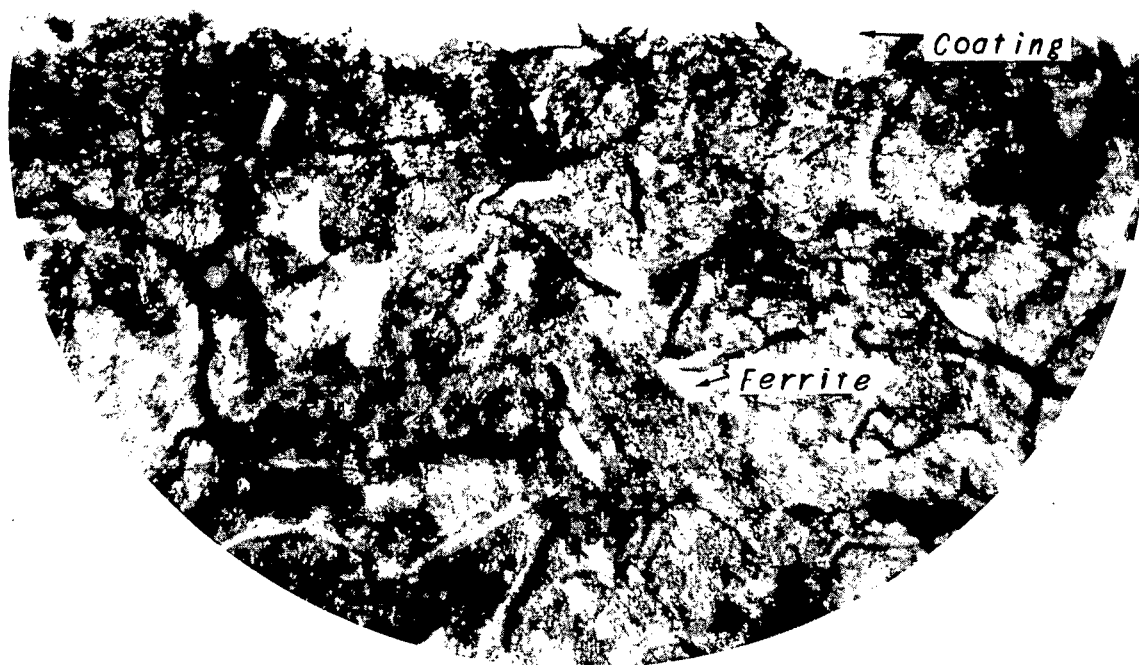


Figure 1. - Transverse section of the running face of a new cast-iron piston ring. Etched in nital. X1500.



*Figure 2. - Plan view of the running face of a scuffed cast-iron piston ring showing ridged condition. Unetched. X35.*

*Nickel plate*



*Figure 3. - Transverse section through scuffed portion of the running face of a cast-iron piston ring showing coating material after test. Etched in nital. X1500.*

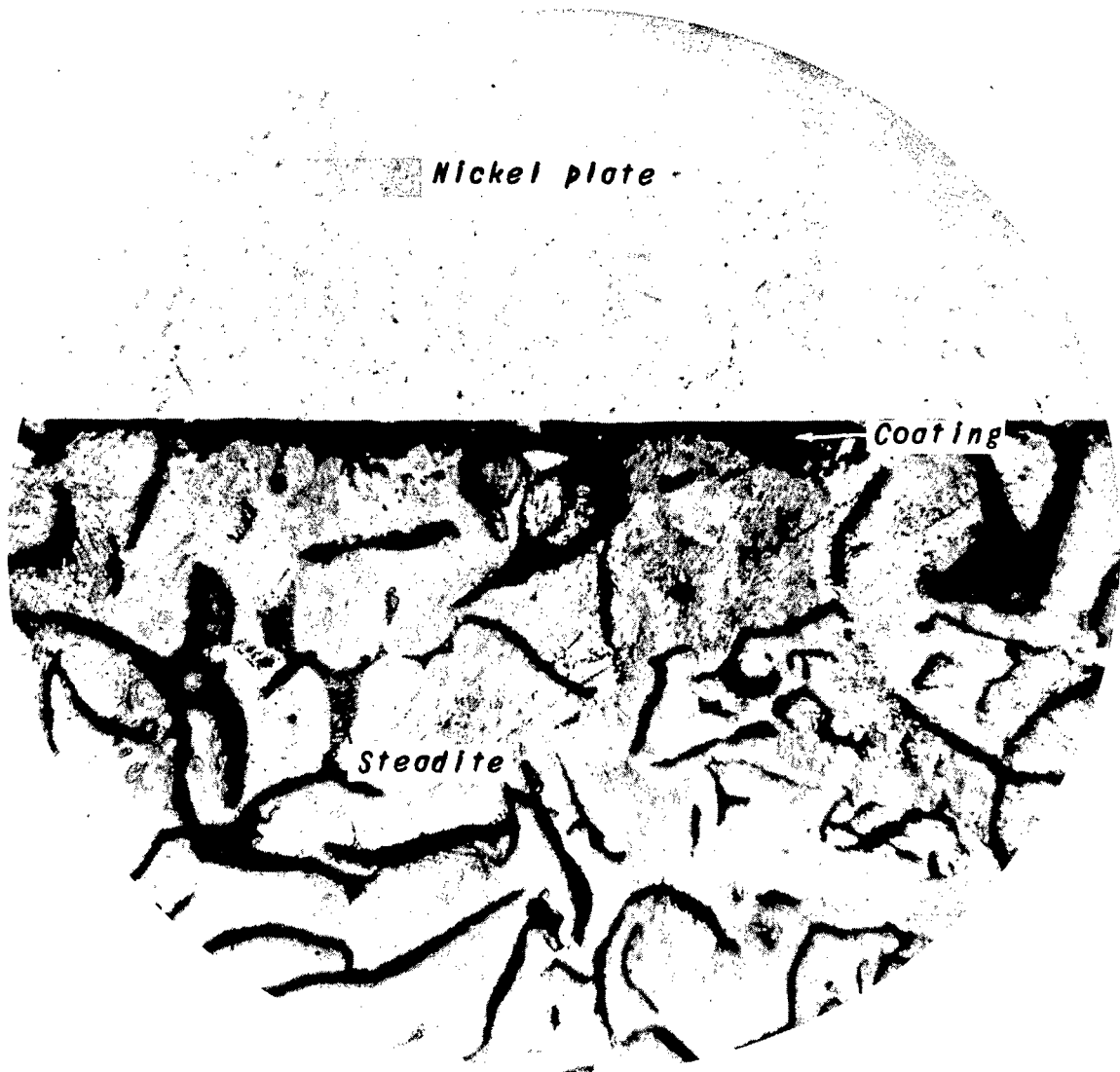


Figure 4. - Transverse section through scuffed portion of the running face of a cast-iron piston ring showing coating material after test. Same area as figure 3. Etched in hot concentrated potassium hydroxide. X1500.

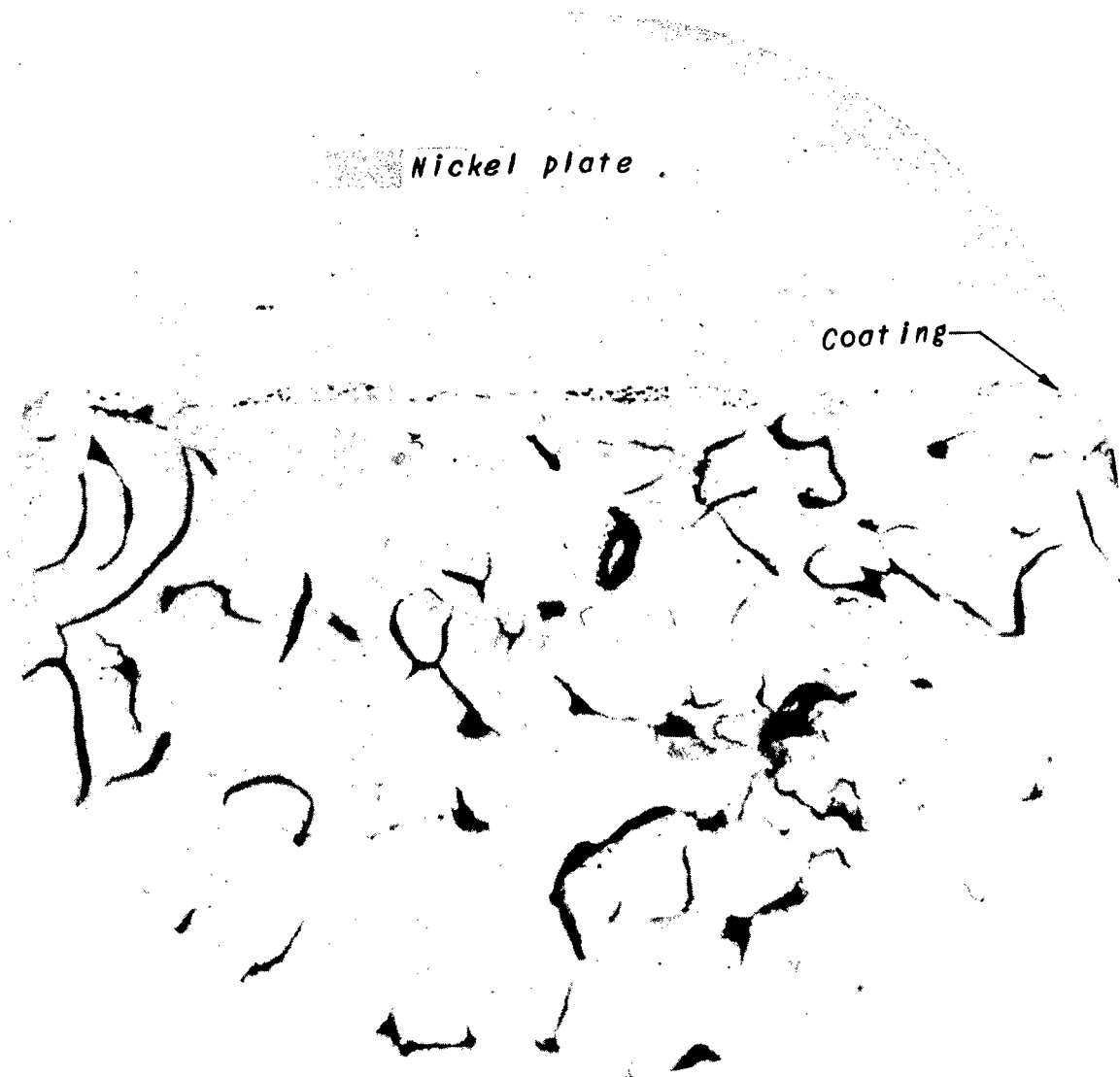


Figure 5. - Transverse section through scuffed portion of the running face of a cast-iron piston ring showing coating after test. Small particles of graphite or voids are visible in the coating material. Unetched. X1500.



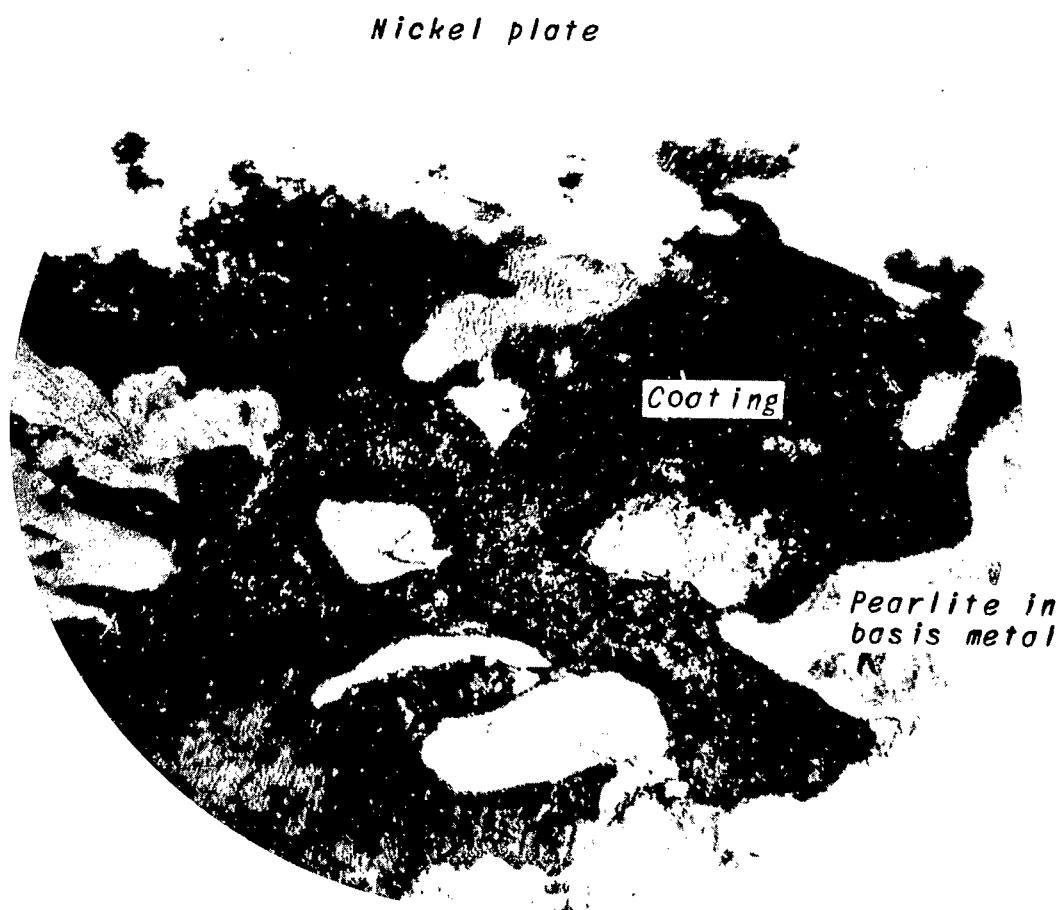
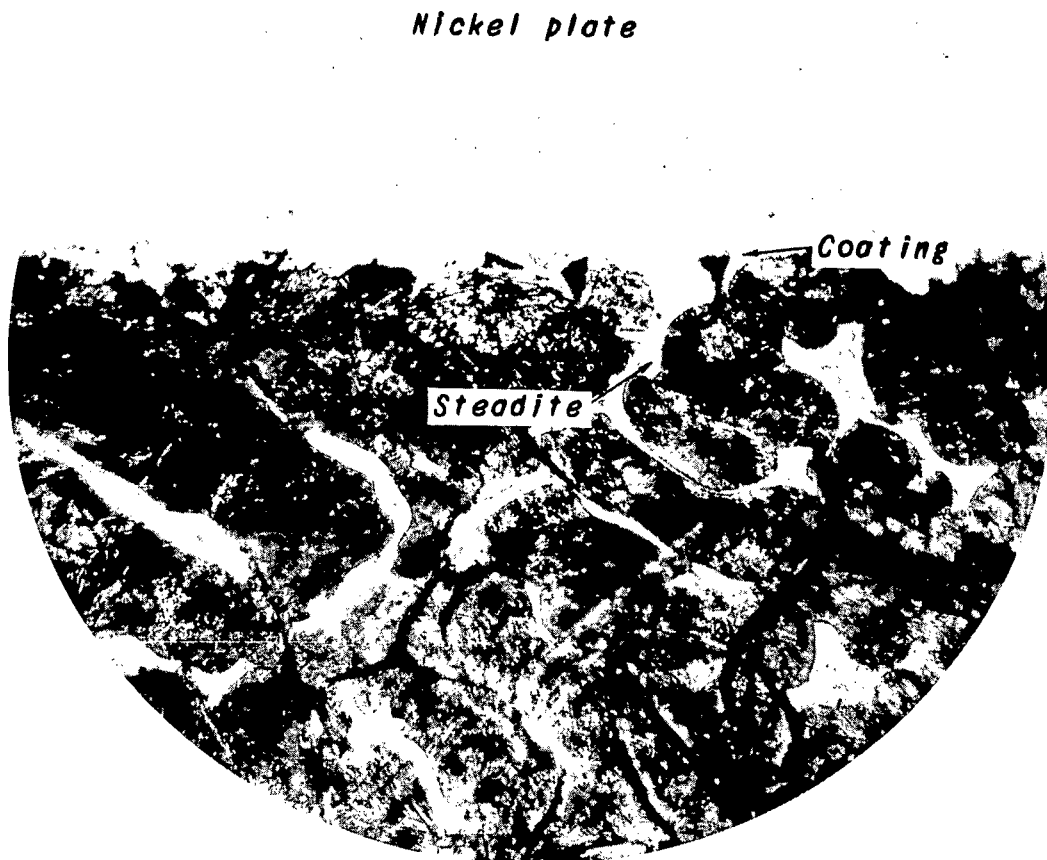


Figure 6. - Taper section through scuffed portion of the running face of a cast-iron piston ring showing coating material after test. Etched in hot concentrated potassium hydroxide; oblique illumination; effective vertical magnification, X37,500; horizontal magnification, X1500.



*Figure 7. - Transverse section through scuffed portion of the running face of a cast-iron piston ring showing coating material after test. Etched in nital. X1500.*

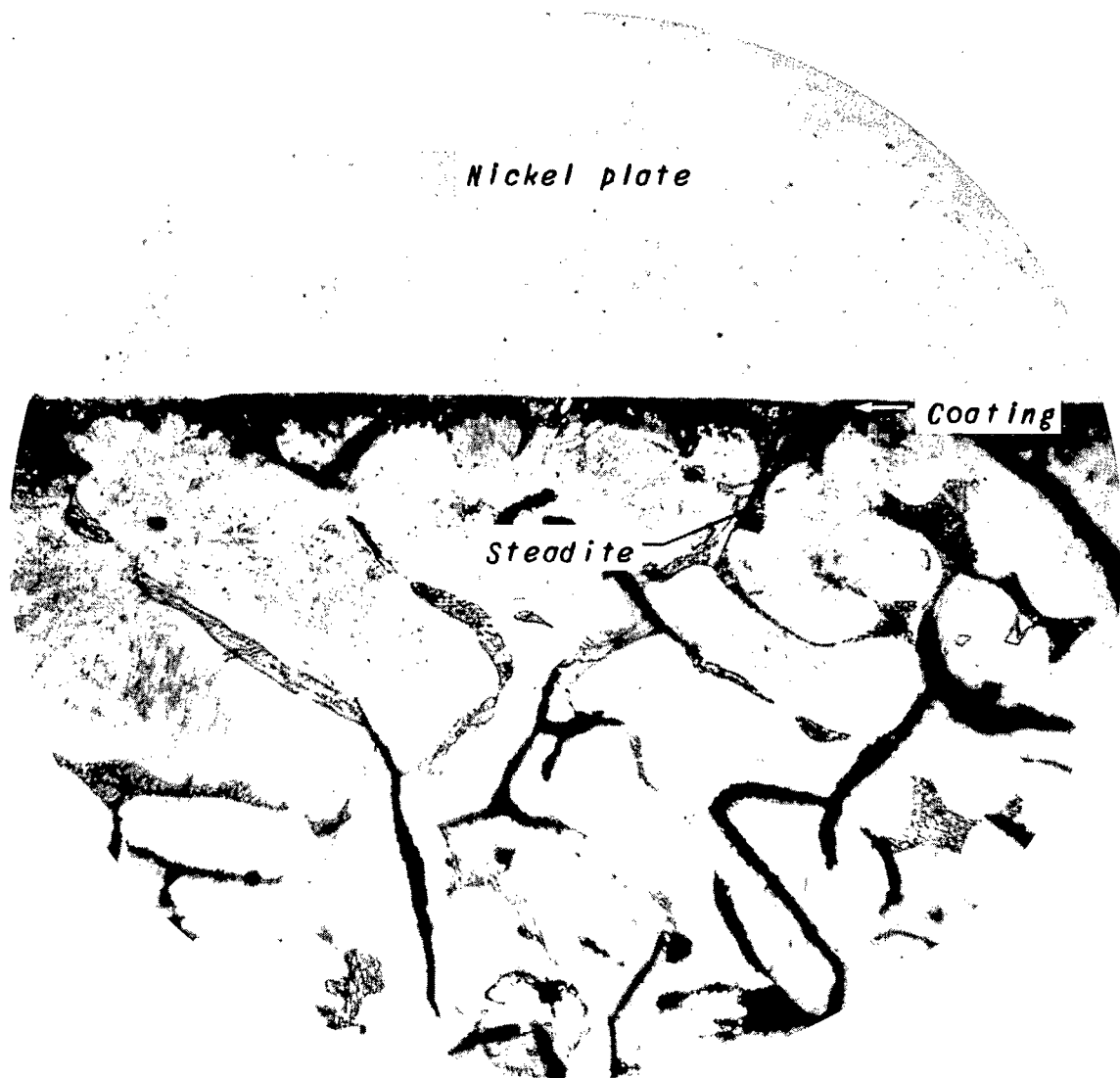
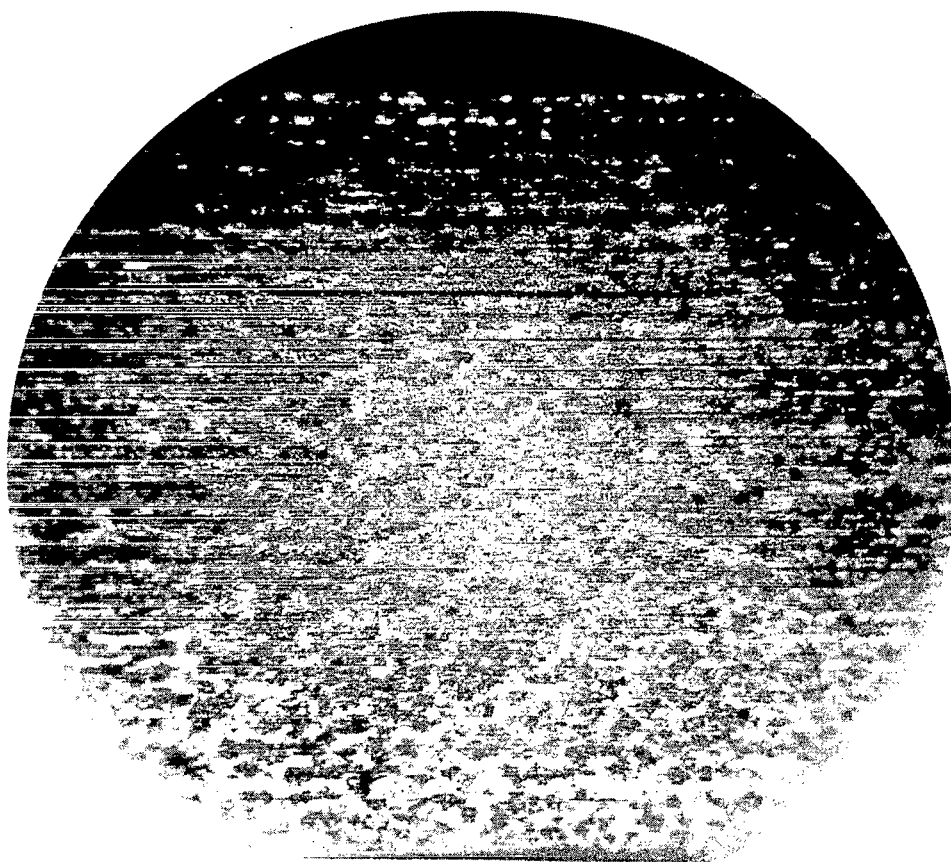
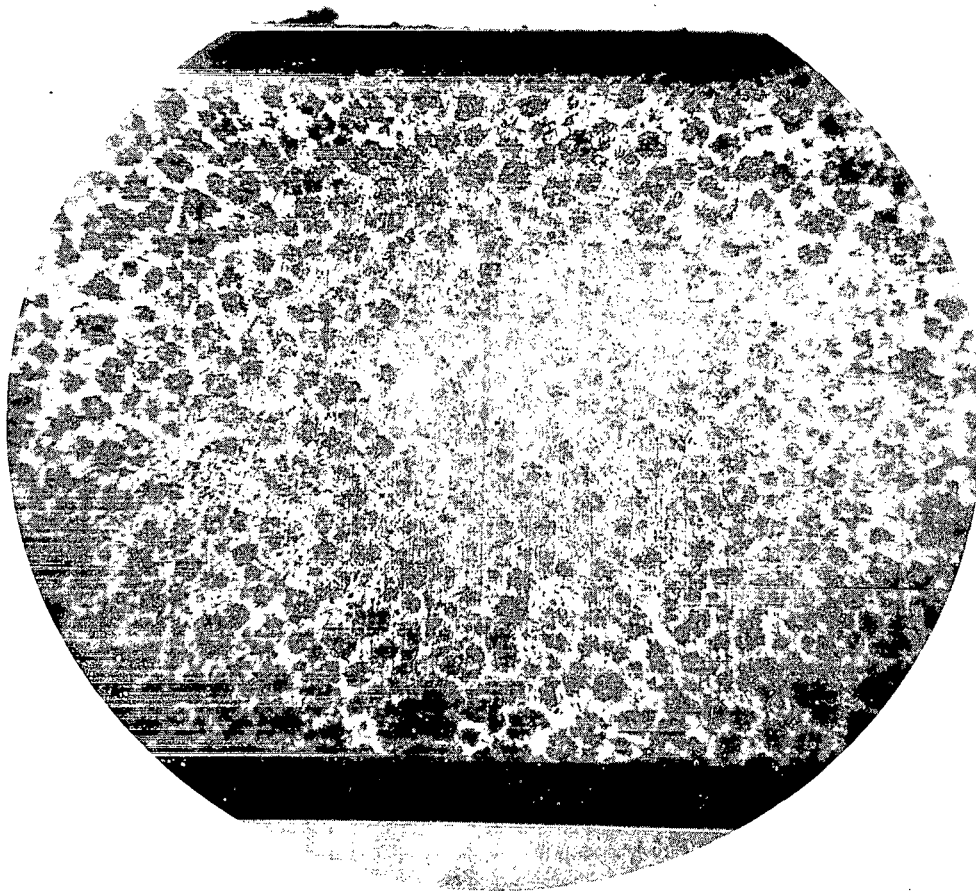


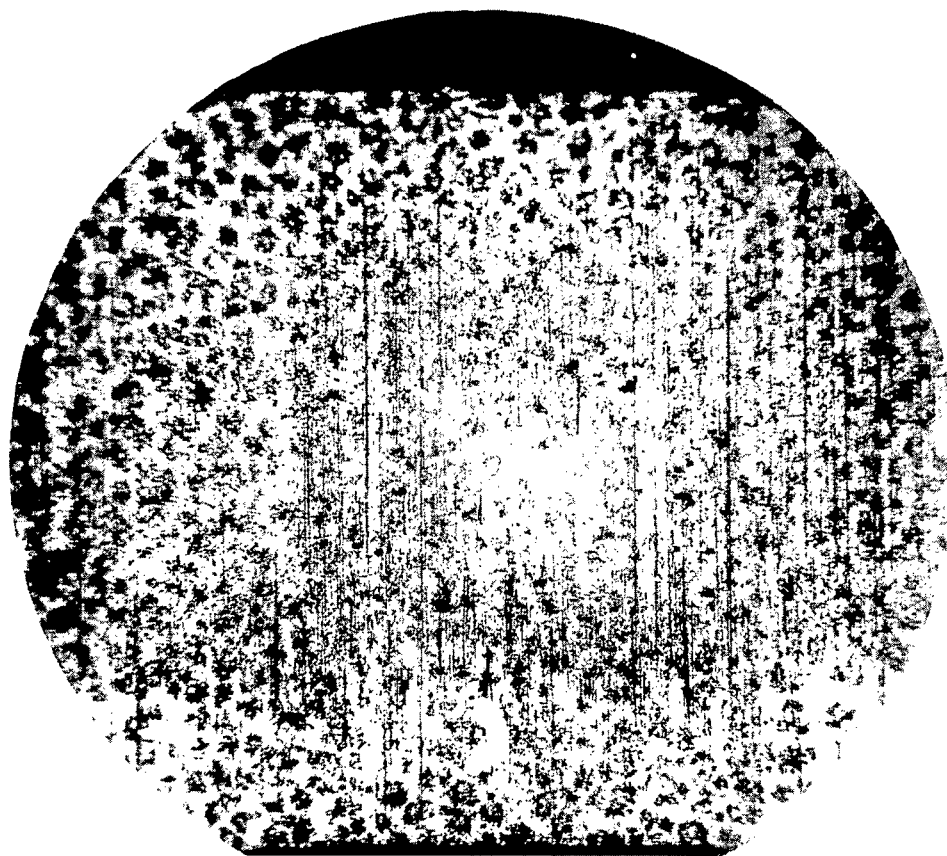
Figure 8. - Transverse section through scuffed portion of a cast-iron piston ring showing coating material after test. Same area as figure 7. Etched in hot concentrated potassium hydroxide. X1500.



*Figure 9. - Running face of a new cast-iron piston ring. The light reflective areas are steadite. Etched with Stead's reagent. X35.*



*Figure 10. - Running face of a normally operated, unscuffed cast-iron piston ring. The light reflective areas are steadite. Etched with Stead's reagent. X35.*



*Figure 11. - Running face of a scuffed cast-iron piston ring. The light reflective areas are steadite. Etched with Stead's reagent. X35.*

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